Pace Analytical Services, Inc.
315 Chestnut Street
Virginia, MN 55792
(218) 742-1042

April 14, 2016

Tom Moe
USS Corporation
P.O. Box 417
Mountain Iron, MN 55768

RE: Project: NPDES-TB Wk1
Pace Project No.: 1263650

Dear Tom Moe:
Enclosed are the analytical results for sample(s) received by the laboratory on April 06, 2016. The results relate only to the samples included in this report. Results reported herein conform to the most current TNI standards and the laboratory's Quality Assurance Manual, where applicable, unless otherwise noted in the body of the report.

If you have any questions concerning this report, please feel free to contact me.

Sincerely,

Melisa M Woods
melisa.woods@pacelabs.com
Project Manager

Enclosures

cc: Terri Sabetti, NTS

Pace Analytical Services, Inc.
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CERTIFICATIONS

Project: NPDES-TB Wk1
Pace Project No.: 1263650

Virginia Minnesota Certification ID's
315 Chestnut Street, Virginia, MN 55792
Alaska Certification #MN01084
Arizona Department of Health Certification #AZ0785
Minnesota Dept of Health Certification #: 027-137-445
North Dakota Certification: # R-203
Wisconsin DNR Certification # : 998027470
WA Department of Ecology Lab ID# C1007
Nevada DNR #MN010842015-1
Oklahoma Department of Environmental Quality

Duluth Minnesota Cerification ID's
4730 Oneota St., Duluth, MN 55807
Minnesota Dept of Health Certification #: 027-137-152
Wisconsin DNR Certification # : 999446800
North Dakota Certification #: R-105

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
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SAMPLE SUMMARY

Project: NPDES-TB Wk1
Pace Project No.: 1263650

Lab ID	Sample ID	Matrix	Date Collected	Date Received
1263650001	**SD 001 (Seep 020)**	Water	04/06/16 11:30	04/06/16 13:50

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SAMPLE ANALYTE COUNT

Project: NPDES-TB Wk1
Pace Project No.: 1263650

Lab ID	Sample ID	Method	Analysts	Analytes Reported	Laboratory
1263650001	**SD 001 (Seep 020)**	EPA 1664 TPH	BT1	1	PASI-DUL
		USGS I-3765	BEM	1	PASI-V
		EPA 300.0	CSD	1	PASI-V

Pace Analytical Services, Inc.
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ANALYTICAL RESULTS

Project: NPDES-TB Wk1
Pace Project No.: 1263650

Sample: SD 001 (Seep 020) **Lab ID: 1263650001** Collected: 04/06/16 11:30 Received: 04/06/16 13:50 Matrix: Water

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
1664 SGT-HEM, TPH	Analytical Method: EPA 1664 TPH								
Total Petroleum Hydrocarbons	ND	mg/L	3.0	0.90	1		04/11/16 15:58		
USGS I-3765 TSS	Analytical Method: USGS I-3765								
Total Suspended Solids	**4.4**	mg/L	1.0	1.0	1		04/08/16 11:51		
300.0 IC Anions 28 Days	Analytical Method: EPA 300.0								
Sulfate	**1060**	mg/L	20.0	0.89	10		04/08/16 03:43	14808-79-8	

REPORT OF LABORATORY ANALYSIS

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QUALITY CONTROL DATA

Project: NPDES-TB Wk1
Pace Project No.: 1263650

QC Batch: DUL/5979
QC Batch Method: EPA 1664 TPH
Analysis Method: EPA 1664 TPH
Analysis Description: 1664 SGT-HEM, TPH
Associated Lab Samples: 1263650001

METHOD BLANK: 304848 Matrix: Water
Associated Lab Samples: 1263650001

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
Total Petroleum Hydrocarbons	mg/L	ND	3.0	0.90	04/11/16 14:02	

LABORATORY CONTROL SAMPLE: 304849

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Total Petroleum Hydrocarbons	mg/L	20	14.4	72	64-132	

MATRIX SPIKE SAMPLE: 304850

Parameter	Units	1263629001 Result	Spike Conc.	MS Result	MS % Rec	% Rec Limits	Qualifiers
Total Petroleum Hydrocarbons	mg/L	<0.92	20.4	13.2	64	64-132	

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

REPORT OF LABORATORY ANALYSIS

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QUALITY CONTROL DATA

Project: NPDES-TB Wk1
Pace Project No.: 1263650

QC Batch: WET/23339
QC Batch Method: USGS I-3765
Analysis Method: USGS I-3765
Analysis Description: USGS I-3765 Total Suspended Solids
Associated Lab Samples: 1263650001

METHOD BLANK: 304412 Matrix: Water
Associated Lab Samples: 1263650001

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
Total Suspended Solids	mg/L	ND	1.0	1.0	04/08/16 11:51	

LABORATORY CONTROL SAMPLE: 304413

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Total Suspended Solids	mg/L	239	222	93	80-120	

SAMPLE DUPLICATE: 304414

Parameter	Units	1263662003 Result	Dup Result	RPD	Max RPD	Qualifiers
Total Suspended Solids	mg/L	158	166	5	10	

SAMPLE DUPLICATE: 304415

Parameter	Units	1263642001 Result	Dup Result	RPD	Max RPD	Qualifiers
Total Suspended Solids	mg/L	184	200	8	10	

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REPORT OF LABORATORY ANALYSIS

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QUALITY CONTROL DATA

Project: NPDES-TB Wk1
Pace Project No.: 1263650

QC Batch: WETA/16211 Analysis Method: EPA 300.0
QC Batch Method: EPA 300.0 Analysis Description: 300.0 IC Anions
Associated Lab Samples: 1263650001

METHOD BLANK: 303898 Matrix: Water
Associated Lab Samples: 1263650001

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
Sulfate	mg/L	ND	2.0	0.089	04/07/16 21:08	

LABORATORY CONTROL SAMPLE: 303899

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Sulfate	mg/L	50	49.6	99	90-110	

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 303900 303901

Parameter	Units	1263656001 Result	MS Spike Conc.	MSD Spike Conc.	MS Result	MSD Result	MS % Rec	MSD % Rec	% Rec Limits	RPD	Max RPD	Qual
Sulfate	mg/L	27.3	250	250	291	290	105	105	90-110	0	20	

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 303902 303903

Parameter	Units	1263575002 Result	MS Spike Conc.	MSD Spike Conc.	MS Result	MSD Result	MS % Rec	MSD % Rec	% Rec Limits	RPD	Max RPD	Qual
Sulfate	mg/L	21.1	250	250	285	285	106	105	90-110	0	20	

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
315 Chestnut Street
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QUALIFIERS

Project: NPDES-TB Wk1
Pace Project No.: 1263650

DEFINITIONS

DF - Dilution Factor, if reported, represents the factor applied to the reported data due to dilution of the sample aliquot.

ND - Not Detected at or above adjusted reporting limit.

J - Estimated concentration above the adjusted method detection limit and below the adjusted reporting limit.

MDL - Adjusted Method Detection Limit.

PQL - Practical Quantitation Limit.

RL - Reporting Limit.

S - Surrogate

1,2-Diphenylhydrazine decomposes to and cannot be separated from Azobenzene using Method 8270. The result for each analyte is a combined concentration.

Consistent with EPA guidelines, unrounded data are displayed and have been used to calculate % recovery and RPD values.

LCS(D) - Laboratory Control Sample (Duplicate)

MS(D) - Matrix Spike (Duplicate)

DUP - Sample Duplicate

RPD - Relative Percent Difference

NC - Not Calculable.

SG - Silica Gel - Clean-Up

U - Indicates the compound was analyzed for, but not detected.

N-Nitrosodiphenylamine decomposes and cannot be separated from Diphenylamine using Method 8270. The result reported for each analyte is a combined concentration.

Pace Analytical is TNI accredited. Contact your Pace PM for the current list of accredited analytes.

TNI - The NELAC Institute.

LABORATORIES

PASI-DUL	Pace Analytical Services - Duluth
PASI-V	Pace Analytical Services - Virginia

Pace Analytical Services, Inc.
315 Chestnut Street
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QUALITY CONTROL DATA CROSS REFERENCE TABLE

Project: NPDES-TB Wk1
Pace Project No.: 1263650

Lab ID	Sample ID	QC Batch Method	QC Batch	Analytical Method	Analytical Batch
1263650001	**SD 001 (Seep 020)**	EPA 1664 TPH	DUL/5979		
1263650001	**SD 001 (Seep 020)**	USGS I-3765	WET/23339		
1263650001	**SD 001 (Seep 020)**	EPA 300.0	WETA/16211		

Pace Analytical

CHAIN-OF-CUSTODY / Analytical Req

The Chain-of-Custody is a LEGAL DOCUMENT. All relevant

WO# : 1263650

PM: MMW Due Date: 04/20/16

CLIENT: USS CORP

1

Section A Required Client Information:		Section B Required Project Information:		Section C Invoice Information:	
Company:	USS Corporation	Report To:	Tom Moe	Attention:	
Address:	P.O. Box 417	Copy To:		Company Name:	
Mt. Iron, MN 55768				Address:	
Email:		Purchase Order #:		Pace Quote:	
Phone:	Fax:	Project Name:	NPDES-TB Wk1	Pace Project Manager:	heather.zika@pacelabs.com,
Requested Due Date:		Project #:		Pace Profile #:	

SAMPLE ID
One Character per box.
(A-Z, 0-9 / , -)
Sample Ids must be unique

MATRIX	CODE
Drinking Water	DW
Water	WT
Waste Water	WW
Product	P
Soil/Solid	SL
Oil	OL
Wipe	WP
Air	AR
Other	OT
Tissue	TS

ITEM #	SAMPLE ID	MATRIX CODE (see valid codes to left)	SAMPLE TYPE (G=GRAB C=COMP)	COLLECTED START DATE	COLLECTED START TIME	COLLECTED END DATE	COLLECTED END TIME	SAMPLE TEMP AT COLLECTION	# OF CONTAINERS	Preservatives: Unpreserved	H2SO4	HNO3	HCl	NaOH	Na2S2O3	Methanol	Other	Analyses Test Y/N: TSS,SO4	TRPH 1664	Residual Chlorine (Y/N)
1	SD 001 (Seep 020)	WT		4-6-16	11:30	4-6-16	11:30											X	X	
2																				
3																				
4																				
5																				
6																				
7																				
8																				
9																				
10																				
11																				
12																				

RELINQUISHED BY / AFFILIATION	DATE	TIME	ACCEPTED BY / AFFILIATION	DATE	TIME	SAMPLE CONDITIONS: TEMP in C	Received on Ice (Y/N)	Custody Sealed Cooler (Y/N)	Samples Intact (Y/N)
Paul Mattila	4-6-16	13:50		4-6-16	1350	3.3	Y	N	Y

SAMPLER NAME AND SIGNATURE

PRINT Name of SAMPLER: Paul Mattila

SIGNATURE of SAMPLER: Paul Mattila

DATE Signed: 4-6-16

Pace Analytical®	Document Name: Sample Condition Upon Receipt Form	Document Revised: 23Feb2015 Page 1 of 1
	Document No.: F-VM-C-001-Rev.09	Issuing Authority: Pace Virginia, Minnesota Quality Office

Sample Condition Upon Receipt

Client Name: USS

Project #: WO# : 1263650

1263650

Courier: ☐ Fed Ex ☐ UPS ☐ USPS ☑ Client ☐ Commercial ☐ Pace ☐ Other: _____

Tracking Number: _____

Custody Seal on Cooler/Box Present? ☐ Yes ☒ No Seals Intact? ☐ Yes ☒ No

Optional: Proj. Due Date: Proj. Name:

Packing Material: ☐ Bubble Wrap ☒ Bubble Bags ☐ None ☐ Other: _____ Temp Blank? ☒ Yes ☐ No

Thermometer Used: ☒ 140792808 Type of Ice: ☒ Wet ☐ Blue ☐ None ☒ Samples on ice, cooling process has begun

Cooler Temp Read °C: 3.0 Cooler Temp Corrected °C: 3.3

Temp should be above freezing to 6°C Correction Factor: 0.3 Biological Tissue Frozen? ☐ Yes ☐ No ☒ NA

Date and Initials of Person Examining Contents: CL 4-6-16

				Comments:
Chain of Custody Present?	☒ Yes	☐ No	☐ N/A	1.
Chain of Custody Filled Out?	☒ Yes	☐ No	☐ N/A	2.
Chain of Custody Relinquished?	☒ Yes	☐ No	☐ N/A	3.
Sampler Name and Signature on COC?	☒ Yes	☐ No	☐ N/A	4.
Samples Arrived within Hold Time?	☒ Yes	☐ No	☐ N/A	5.
Short Hold Time Analysis (<72 hr)?	☐ Yes	☒ No	☐ N/A	6.
Rush Turn Around Time Requested?	☐ Yes	☒ No	☐ N/A	7.
Sufficient Volume?	☒ Yes	☐ No	☐ N/A	8.
Correct Containers Used?	☒ Yes	☐ No	☐ N/A	9.
-Pace Containers Used?	☒ Yes	☐ No	☐ N/A	
Containers Intact?	☒ Yes	☐ No	☐ N/A	10.
Filtered Volume Received for Dissolved Tests?	☐ Yes	☐ No	☒ N/A	11. Note if sediment is visible in the dissolved containers.
Sample Labels Match COC?	☒ Yes	☐ No	☐ N/A	12.
-Includes Date/Time/ID/Analysis Matrix: WT				
All containers needing acid/base preservation will be checked and documented in the pH logbook.	☐ Yes	☐ No	☒ N/A	See pH log for results and additional preservation documentation
Headspace in Methyl Mercury Container	☐ Yes	☐ No	☒ N/A	13.
Headspace in VOA Vials (>6mm)?	☐ Yes	☐ No	☒ N/A	14.
Trip Blank Present?	☐ Yes	☐ No	☒ N/A	15.
Trip Blank Custody Seals Present?	☐ Yes	☐ No	☒ N/A	
Pace Trip Blank Lot # (if purchased): _____				

CLIENT NOTIFICATION/RESOLUTION

Field Data Required? ☐ Yes ☐ No

Person Contacted: _____ Date/Time: _____

Comments/Resolution: _____

FECAL WAIVER ON FILE Y N TEMPERATURE WAIVER ON FILE Y N

Project Manager Review: KAH for MMW Date: 4-6-16

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e out of hold, incorrect preservative, out of temp, incorrect containers)

Intra-Regional Chain of Custody

Pace Analytical
www.pacelabs.com

Workorder: 1263650 **Workorder Name:** NPDES-TB Wk1 **Owner Received Date:** 4/6/2016 **Due Date:** 4/20/2016

Received at:	Send To Lab:
Pace Analytical Virginia 315 Chestnut Street Virginia, MN 55792 Phone (218) 742-1042	Pace Analytical Duluth 4730 Oneota Street Duluth, MN 55807 Phone (218) 727-6380

Report To:
Melisa M Woods

Item	Sample ID	Sample Type	Collect Date/Time	Lab ID	Matrix	Preserved Containers: HCL	Requested Analysis: EPA 1664 TPH	LAB USE ONLY
1	SD 001 (Seep 020)	PS	4/6/2016 11:30	1263650001	Water	3	X	
2								
3								
4								
5								

Transfers	Released By	Date/Time	Received By	Date/Time
1		4/7/16 1[illegible]	Kristina Polson	4/7 1520
2				
3				
4				

Comments

Cooler Temperature on Receipt 4.1 °C | **Custody Seal** Y or N | **Received on Ice** Y or N | **Samples Intact** Y or N

****In order to maintain client confidentiality, location/name of the sampling site, sampler's name and signature may not be provided on this COC document. This chain of custody is considered complete as is since this information is available in the owner laboratory.*

Pace Analytical®	Document Name: Sample Condition Upon Receipt Form	Document Revised: 22Jan2016 Page 1 of 1
	Document No.: F-DUL-C-001-Rev.01	Issuing Authority: Pace Virginia, Minnesota Quality Office

Sample Condition Upon Receipt

Client Name: IR-COC

Project #:

Courier: ☐ Fed Ex ☐ UPS ☐ USPS ☐ Client ☐ Commercial ☐ Pace ☒ Other: Jake

Tracking Number:

Custody Seal on Cooler/Box Present? ☒ Yes ☐ No Seals Intact? ☒ Yes ☐ No

Optional: Proj. Due Date: Proj. Name:

Packing Material: ☐ Bubble Wrap ☒ Bubble Bags ☐ None ☐ Other: Temp Blank? ☒ Yes ☐ No

Thermometer Used: ~~☒ B00051~~ 4/7/16 KP IR-1 Type of Ice: ☒ Wet ☐ Blue ☐ None ☒ Samples on ice, cooling process has begun

Cooler Temp Read °C: 3.5 Cooler Temp Corrected °C: 4.1 Biological Tissue Frozen? ☐ Yes ☐ No ☒ NA

Temp should be above freezing to 6°C Correction Factor: +0.6 °C Date and Initials of Person Examining Contents: 4/7/16 KP

Comments:

Chain of Custody Present?	☒ Yes	☐ No	☐ N/A	1.
Chain of Custody Filled Out?	☒ Yes	☐ No	☐ N/A	2.
Chain of Custody Relinquished?	☒ Yes	☐ No	☐ N/A	3.
Sampler Name and Signature on COC?	☐ Yes	☐ No	☒ N/A	4.
Samples Arrived within Hold Time?	☒ Yes	☐ No	☐ N/A	5.
Short Hold Time Analysis (<72 hr)?	☐ Yes	☒ No	☐ N/A	6.
Rush Turn Around Time Requested?	☐ Yes	☒ No	☐ N/A	7.
Sufficient Volume?	☒ Yes	☐ No	☐ N/A	8.
Correct Containers Used?	☒ Yes	☐ No	☐ N/A	9.
-Pace Containers Used?	☒ Yes	☐ No	☐ N/A	
Containers Intact?	☒ Yes	☐ No	☐ N/A	10.
Filtered Volume Received for Dissolved Tests?	☐ Yes	☐ No	☒ N/A	11. Note if sediment is visible in the dissolved containers.
Sample Labels Match COC?	☒ Yes	☐ No	☐ N/A	12.
-Includes Date/Time/ID/Analysis Matrix: WT				
All containers needing acid/base preservation will be checked and documented in the pH logbook.	☐ Yes	☐ No	☐ N/A	See pH log for results and additional preservation documentation
Headspace in Methyl Mercury Container	☐ Yes	☐ No	☐ N/A	13.
Headspace in VOA Vials (>6mm)?	☐ Yes	☐ No	☐ N/A	14.
Trip Blank Present?	☐ Yes	☐ No	☐ N/A	15.
Trip Blank Custody Seals Present?	☐ Yes	☐ No	☐ N/A	
Pace Trip Blank Lot # (if purchased):				

CLIENT NOTIFICATION/RESOLUTION Field Data Required? ☐ Yes ☐ No

Person Contacted: Date/Time:

Comments/Resolution:

FECAL WAIVER ON FILE Y N TEMPERATURE WAIVER ON FILE Y N

Project Manager Review: UMF Date: 4-7-16

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e out of hold, incorrect preservative, out of temp, incorrect containers)